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Microlaminate composites with two-dimensional strengthening have been successfully fabricated using reactive ion plating and hot pressing. The composite materials have been mechanically tested and characterized structurally and chemically. It has been observed that these laminate composites can be carefully designed to exhibit strengthening much greater than that predicted by the simple rule-of-mixtures and still maintain reasonable ductility. However, composite strengthening is limited by reinforcement precracking, diffusion bond delamination, and matrix/reinforcement reaction.

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From the results of this research, a fundamental understanding of these engineered composite materials has been developed and specifically the relationships between: 1) ion plating plating and vacuum hot press processing parameters; 2) microstructure and microchemistry of the composite and interfaces; and 3) mechanical behavior and the micromechanics of deformation and fracture.

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# STRUCTURE AND PROPERTIES OF TITANIUM NITRIDE STRENGTHENED MICROLAMINATE METAL MATRIX COMPOSITES

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THESIS
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for the degree of Doctorate of Philosophy in Metallurgical Engineering
in the Graduate College of the
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## PROGRAM SUMMARY

This investigation examined the structure and properties of titanium nitride strengthened microlaminate metal matrix composites. Reactive ion plated titanium nitride (TiN) was deposited onto titanium, nickel, and aluminum foils which were subsequently hot pressed to form microlaminate composites. The TiN volume percent was varied and its effect upon the structure and properties of the composites investigated. Scanning electron microscopy (SEM), cross-sectional transmission electron microscopy (TEM) and scanning Auger electron spectroscopy (AES) have been used to characterize the film morphology, the fracture behavior of the composites, and the nature of the metal/ceramic interfaces formed during reactive ion plating and hot pressing. The mechanical properties (ultimate tensile strength, strain-to-failure and elastic modulus) of the microlaminate composites were determined by tensile testing specimens machined from the hot pressed composites.

The TiN strengthened Al matrix microlaminate composites exhibited poor mechanical properties due to reinforcement precracking (due to thermal expansion mismatch and weak intercolumnar boundary

strength of TiN film) and poor diffusion bonding both of which promoted failure of the hot pressed metal/ceramic interface at low applied loads. TEM examination of the Al/TiN composite interfaces showed the ion plated interface to be fully dense and continuous while the hot pressed interface exhibited porosity and mechanical interlocking of the Al and TiN surfaces (poor bonding). Interfacial modification of the Al-TiN hot pressed bond by the addition of a Ti film overlayer ion plated on the TiN film improved the mechanical behavior of the TiN strengthened Al matrix composite, by increasing the interfacial adherance between the Al matrix and TiN film reinforcement.

The TiN strengthened Ni matrix microlaminate composites exhibited strengths predicted by rule-of-mixtures calculations (ROM) which indicated that sufficient bonding between the TiN reinforcement and Ni matrix was present to allow the transfer of the applied load from the matrix to the reinforcement. XTEM analysis of the Ni/TiN composite interfaces showed both the ion plated and hot pressed interfaces to be well adhered and continuous. The addition of a Ti film overlayer ion plated on the TiN film improved the strength of the composite by altering the nature of the interfaces present as a result of the formation of the $Ni_3Ti$ intermetallic phase.

The TiN strengthened Ti matrix microlaminate composites exhibited strengths greater than that predicted by rule-of-mixtures calculations while maintaining some ductility. However, after the initial few volume percent of TiN, the mechanical response of the composites diminished due to the increased crack sensitivity of the Ti matrix and the delamination of the interface region both of which are the result of the dissolution of the TiN film by the Ti matrix during the hot pressing procedure. The delamination of the interface region is greatly assisted by the connectivity of voids present in the dissolved TiN zone. TEM examination of the Ti/TiN composite interfaces showed the dissolution of the TiN film by the Ti matrix, the reduction of TiN to $Ti_2N$ and Ti, and the presence of voids in the transformed film region due to the diffusion of nitrogen from the film to the matrix.

## RESEARCH CONCLUSIONS

Microlaminate composites with two-dimensional strengthening have been successfully fabricated using reactive ion plating and hot pressing. The composite materials have been mechanically tested and characterized structurally and chemically. It has been observed that these laminate composites can be carefully designed to exhibit strengthening much greater than that predicted by the simple rule-of-mixtures and still maintain reasonable ductility. However, composite strengthening is limited by reinforcement precracking, diffusion bond delamination, and matrix/reinforcement reaction.

From the results of this research, a fundamental understanding of these engineered composite materials has been developed and specifically the relationships between: 1) ion plating plating and vacuum hot press processing parameters; 2) microstructure and microchemistry of the composite and interfaces; and 3) mechanical behavior and the micromechanics of deformation and fracture. Based upon the work of this research program the following statements have been outlined:

1. Ion plating is an effective technique for producing novel metal/ceramic microlaminate composite materials by virtue of its ability to deposit controlled volume percents of film reinforcement and produce highly adherent interfaces.

   *The ion plating process can deposit a metal overlayer (Ti) on the ceramic film reinforcement (TiN) thereby changing the bonding characteristics of the various microlaminate composites.*

2. The ion plated interfaces and hot pressed interfaces differ structurally and chemically as shown by XRD, AES, and XTEM analysis.

   *The ion plated interface was shown to be fully dense and continuous along the film/matrix (e.g. TiN/Al) and*

*film/film (Ti/TiN) interfaces. The hot pressed Al-TiN interface was shown by XTEM to be very irregular and contain internal flaws and voids.*

3. The mechanical integrity of the hot pressed bonds of these composites is very sensitive to the interdiffusion characteristics of the materials to be joined and the resulting interfacial defects.

   *XTEM analysis of the Al/TiN-Al and Ni/TiN-Ni composites revealed that the Al-TiN hot pressed interface consisted of a mechanical bonding which resulted in the non-existent strengthening effect imparted by the TiN to the Al and that the Ni-TiN interface was well adhered and continuous which was reflected in the TiN strengthening of the Ni matrix.*

4. The laminate structure provided favorable strengthening while maintaining reasonable ductility. When failure occurred, it was a complicated mixed-mode failure involving such fracture mechanisms as brittle fracture of the filler, necking of the matrix, delamination along the hot pressed and ion plated interfaces, cracking of the reinforcement, and secondary crack initiation and propagation.

5. The mechanical behavior of these microlaminate materials does not conform to a conventional theory such as "rule-of-mixtures" but this theory does give a lower bound approximation.

The fundamental knowledge gained from the examination of these specific composites leads to the following basic conclusions for composite materials.

- Processing techniques can be optimized for the desired material characteristics.
  *Careful choice of composite materials: Ni/TiN-Ti*

- Modification of the nature of the composite interfaces is directly observed by the mechanical response of the composite material.
  *Addition of Ti film overlayer: Al/TiN/Ti-Al*

- Interface reactions between the matrix and reinforcement can enhance or reduce the physical properties of the composite.
  *Extent of reaction layer: Ni/Ti-Ni and Ni/TiN/Ti-Ni*

- Chemical compatibility between the matrix and reinforcement is critical for proper composite properties.
  *Dissolution of reinforcement: Ti/TiN-Ti*

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